FORM-PTO-1390 (Rev. 12-29-99) U.S. DEPARTMENT OF COMMERCE, PATENT AND TRADEMARK OFFICE

# TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

| ATTORNEY'S DOCKET NUMBER | |
|---|---|
| 010315-152 | |
| U.S. APPLICATION NO (If known, see 37 C.F.R. 1.5) | |
| Unknown 09/856961 | |

| INTERNATIONAL APPLICATION NO. | INTERNATIONAL FILING DATE | PRIORITY DATE CLAIMED |
|---|---|---|
| PCT/SE99/02170 | November 24, 1999 | May 26, 2000 |

TITLE OF INVENTION
A METHOD OF CLEANING SULFIDE CONTAMINATED CONDENSATES

APPLICANT(S) FOR DO/EO/US
Kent K. SANDQUIST and Olle WENNBERG

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and the PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☒ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

**Items 11. to 16. below concern other document(s) or information included:**

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
    ☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:

ABSTRACT on a separate sheet.

531 Rec'd PCT 29 MAY 2001

| U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.5) | INTERNATIONAL APPLICATION NO. | ATTORNEY'S DOCKET NUMBER |
|---|---|---|
| Unknown 09/856961 | PCT/SE99/02170 | 010315-152 |

| 17. ☐ The following fees are submitted: | CALCULATIONS | PTO USE ONLY |
|---|---|---|
| **Basic National Fee (37 CFR 1.492(a)(1)-(5)):** | | |
| Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... $1,000.00 (960) | | |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... $860.00 (970) | | |
| International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... $710.00 (958) | | |
| International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... $690.00 (956) | | |
| International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) ..... $100.00 (962) | | |
| **ENTER APPROPRIATE BASIC FEE AMOUNT =** | $ 1,000.00 | |
| Surcharge of **$130.00 (154)** for furnishing the oath or declaration later than 20 ☐ 30 ☒ months from the earliest claimed priority date (37 CFR 1.492(e)). | $ 130.00 | |

| Claims | Number Filed | Number Extra | Rate | | |
|---|---|---|---|---|---|
| Total Claims | 11 -20 = | 0 | X$18.00 (966) | $ 0 | |
| Independent Claims | 1 -3 = | 0 | X$80.00 (964) | $ 0 | |
| Multiple dependent claim(s) (if applicable) | | | +$270.00 (968) | $ 0 | |

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|---|---|---|
| **TOTAL OF ABOVE CALCULATIONS =** | $ 1,130.00 | |
| Reduction for 1/2 for filing by small entity, if applicable (see below). | $ 565.00 | - |
| **SUBTOTAL =** | $ 565.00 | |
| Processing fee of **$130.00 (156)** for furnishing the English translation later than 20 ☐ 30 ☐ months from the earliest claimed priority date (37 CFR 1.492(f)). + | $ | |
| **TOTAL NATIONAL FEE =** | $ 565.00 | |
| Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00 (581)** per property + | $ | |
| **TOTAL FEES ENCLOSED =** | $ 565.00 | |
| | **Amount to be: refunded** | $ |
| | **charged** | $ |

a. ☐ Small entity status is hereby claimed.

b. ☐ A check in the amount of $____ to cover the above fees is enclosed.

c. ☒ Please charge my Deposit Account No. 02-4800 in the amount of $ 565.00 to cover the above fees. A duplicate copy of this sheet is enclosed.

d. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-4800. A duplicate copy of this sheet is enclosed.

**NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.**

SEND ALL CORRESPONDENCE TO:

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24,970
REGISTRATION NUMBER

Patent
Attorney's Docket No. 010315-152

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

| | | |
|---|---|---|
| In re Patent Application of | ) | |
| | ) | |
| Kent K. SANDQUIST et al | ) | Group Art Unit: Unassigned |
| | ) | |
| Application No.: Unassigned | ) | Examiner: Unassigned |
| | ) | |
| Filed: May 29, 2001 | ) | |
| | ) | |
| For: A METHOD OF CLEANING SULFIDE CONTAMINATED CONDENSATES | ) | |

**PRELIMINARY AMENDMENT**

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Prior to examination on the merits, kindly amend the above-identified application as follows:

**IN THE CLAIMS:**

Kindly replace Claims 1-11, as follows.

1. (Amended) A method of removing sulfides and other volatile contaminants from liquor vapor condensate from a pulp manufacturing process, wherein the said liquor vapor condensate is fed into a stripper, which is part of a closed loop comprising said stripped and a regenerative thermal oxidization process (RTO), in which loop a gas, primarily consisting of air and such components formed or stripped off in the loop, is

circulated, and where the circulating gas is stripping off sulfides and other volatile components from the liquor vapor condensate, whereafter the gas stream is fed into a RTO-process, where the stripped off components are combusted under formation of $SO_2$, and thereafter is the $SO_2$ enriched gas fed either to a $SO_2$ scrubber, where preferably alkali is used as absorption medium, whereafter the circulating gas is returned to the stripper.

2. (Amended) A method as claimed in Claim 1, wherein the $SO_2$ scrubber is part of the closed loop.

3. (Amended) A method as claimed in Claim 1, wherein a minor portion of the gas is bled off from the loop, at the same time air or some other oxygen containing gas is supplied, to ensure that sufficient oxygen is present to safeguard that the oxidization in the RTO-process takes place.

4. (Amended) A method as claimed in Claim 1, wherein the alkali used as absorption medium is oxidized white liquor.

5. (Amended) A method as claimed in Claim 1, wherein the degree of acidification in the $SO_2$ scrubber is controlled to ensure sufficient amount of $SO_2$ remaining in the gas when it is returned to the stripper, wherein $SO_2$ acidifies the

condensate and thereby contributes to enhance the stripping off of sulfides from the condensate.

6. (Amended) A method as claimed in Claim 1, wherein a heat exchanger is installed at a suitable place in the closed loop, to recover or supply energy and thereby to control the temperature in the system.

7. (Amended) A method as claimed in Claim 1, wherein the amount of recirculated gas versus the amount of condensate is controlled for the purpose of optimizing the methanol content in the condensate.

8. (Amended) A method as claimed in Claim 7, wherein such condensate is used as process water in the bleach plant to reduce the bleaching chemical cost.

9. (Amended) A method as claimed in Claim 1, wherein the gas being bled off from the system is minimized by using pure oxygen or an oxygen enriched air mixture, necessary as make up gas for the oxidation.

10. (Amended) A method as claimed in Claim 1, wherein the bled off gas from the system is scrubbed with regard to $SO_2$ in a separate scrubber, which is made up of several absorption steps.

11. (Amended) A method as claimed in Claim 1, wherein the $SO_2$ level is raised to such a level in the system that the absorption medium in the $SO_2$ scrubber gets sufficient acidic, so that this fluid can be utilized as acidification agent in other areas of the pulp mill, or the bleach plant or the tall oil plant.

## REMARKS

By the present Preliminary Amendment, Applicants have amended the claims to remove the multiple dependency and to otherwise place the claims in conformance with U.S. practice. Further attached is an Abstract on a separate sheet.

Entry of the foregoing and prompt and favorable consideration of the subject application on the merits are respectfully requested.

If there are any questions concerning this paper or the application in general, the Examiner is invited to telephone the undersigned at his or her earliest convenience.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

By: _____

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Date: May 29, 2001

**Attachment to Preliminary Amendment dated May 29, 2001**

**Marked-up Claims 1 through 11.**

1. (Amended) A method of removing [sulphides] sulfides and other volatile contaminants from liquor vapor condensate from a pulp manufacturing process, [characterized therein, that] wherein the said liquor vapor condensate is fed into a stripper [(1)], which is part of a closed loop comprising said stripped [(1)] and a regenerative thermal oxidization process (RTO) [(2)], in which loop a gas [(4)], primarily consisting of air and such components formed or stripped off in the loop, is circulated, and where the circulating gas is stripping off [sulphides] sulfides and other volatile components from the liquor vapor condensate [(5)], whereafter the gas stream [(6)] is fed into a RTO-process [(2)], where the stripped off components are combusted under formation of $SO_2$, and thereafter is the $SO_2$ enriched gas [(7)] fed either to a $SO_2$ scrubber [(3)], where preferably alkali is used as absorption medium [(8)], whereafter the circulating gas is returned to the stripper [(1)].

2. (Amended) A method as claimed in Claim 1, [characterized in, that] wherein the $SO_2$ scrubber [(3)] is part of the closed loop.

**Attachment to Preliminary Amendment dated May 29, 2001**

**Marked-up Claims 1 through 11.**

3. (Amended) A method as claimed in [claims 1 or 2, characterized in, that] Claim 1, wherein a minor portion of the gas [(10)] is bled off from the loop, at the same time air or some other oxygen containing gas [(9)] is supplied, to ensure that sufficient oxygen is present to safeguard that the oxidization in the RTO-process [(2)] takes place.

4. (Amended) A method as claimed in [anyone of the preceding claims, characterized in, that] Claim 1, wherein the alkali [(8)] used as absorption medium is oxidized white liquor.

5. (Amended) A method as claimed in [anyone of the preceding claims, characterized in, that] Claim 1, wherein the degree of acidification in the $SO_2$ scrubber [(3)] is controlled to ensure sufficient amount of $SO_2$ remaining in the gas [(4)] when it is returned to the stripper [(1§)], wherein $SO_2$ acidifies the condensate [(5)] and thereby contributes to enhance the stripping off of [sulphides] sulfides from the condensate.

**Attachment to Preliminary Amendment dated May 29, 2001**

**Marked-up Claims 1 through 11.**

6. (Amended) A method as claimed in Claim 1, [characterized in, that] wherein a heat exchanger is installed at a suitable place in the closed loop, to recover or supply energy and thereby to control the temperature in the system.

7. (Amended) A method as claimed in Claim 1, [characterized in, that] wherein the amount of recirculated gas versus the amount of condensate is controlled for the purpose of optimizing the methanol content in the condensate.

8. (Amended) A method as claimed in Claim 7, [characterized in, that] wherein such condensate is used as process water in the bleach plant to reduce the bleaching chemical cost.

9. (Amended) A method as claimed in Claim 1, [characterized in, that] wherein the gas [(10)] being bled off from the system is minimized by using pure oxygen or an oxygen enriched air mixture, necessary as make up gas [(9)] for the oxidation.

**Attachment to Preliminary Amendment dated May 29, 2001**

**Marked-up Claims 1 through 11.**

10. (Amended) A method as claimed in [claims 1 or 2, characterized in, that] Claim 1, wherein the bled off gas [(10)] from the system is scrubbed with regard to $SO_2$ in a separate scrubber, which [preferably] is made up of several absorption steps.

11. (Amended) A method as claimed in [anyone of the preceding claims, characterized in, that] Claim 1, wherein the $SO_2$ level is raised to such a level in the system that the absorption medium in the $SO_2$ scrubber gets sufficient acidic, so that this fluid can be utilized as acidification agent in other areas of the pulp mill, [e.g.,] or the bleach plant or the tall oil plant.

# A method of cleaning sulfide contaminated condensates

In producing chemical pulp according to the Kraft chemical pulp process, waste liquor is produced that is being evaporated prior to burning. During the evaporation process, liquor vapor is stripped off, which in addition to water vapor, also contains certain volatile contaminants. Such contaminants are hydrogen sulfide, methylmercaptan, dimethylsulfide, methanol, terpenes etc. At the evaporation which takes place as a so called multiple effect evaporation with a number of stages, effects (normally 4 - 7), the liquor vapor is also condensed in multiple stages, whereby also large amounts of the volatile contaminants will condense. The condensation takes place in at least as many stages there are effects. This means that the quality of the condensate varies significantly from the different stages of the evaporation. Normally 2-3 different condensate qualities are being separated, where each one is a mixture of condensates from a number of effects. The dirtiest condensate, ( foul condensate), is normally treated in a steam stripper where the volatile components are flashed off. This foul condensate is typically a small amount of the total condensate flow and therefore the steam economy is not affected to any higher degree of the fact that steam is used as the stripper gas. The investment cost can also be kept at a minimum.

The purity of the other condensate qualities is highly dependent on the amount of foul condensate. If the amount of foul condensate is increased the contaminated condensates will be cleaner. A too high amount of foul condensate however the operating and investment cost for the steam stripper system will increase.

The other, less contaminated condensates can to a limited extent be used as process water in dependency of their cleanliness. However if the condensate is too contaminated it can not be re-used but must instead be discharged to the recipient subsequent to some form of treatment.

The primary limiting factor for the use of the contaminated condensate as process water is the content of sulfides, as these can give an unpleasant smell and taste to the pulp. It also creates a significant problem for the working environment. Also terpenes give a smell.

The terpenes however are normally present at very low amounts in the less contaminated condensates.

The technology available to clean these condensates is predominately steam stripping. Since the various condensate flows are very large, the size of the stripper will be significant and a large amount of steam will be required for stripping. The steam volumes will be so large that it will definitely not be economical to use fresh steam. On the other hand it is possible to use flash steam driven off from the evaporation of the waste liquor, in multiple effect evaporation for the stripping. The steam leaving the stripper then can be regained as heat in the next evaporation effect. The cleaning efficiency of such a stripper is however limited since the flash steam from the preceding effect is already contaminated with sulfides, which limits the degree of purity of the output condensate. Primarily the cleanliness is limited regarding sulphides, as the waste liquor can have a considerable content of sulphides. This sulphide content is dependent on that steam is normally taken from the first effect, where the temperature is rather high, which gives an increased sulphide content.

Another drawback is that when the steam passes through the stripper, it loses pressure and volatile components are enriched. These two things will reduce the condensation temperature, which means that the temperature difference available at the evaporation is reduced. The energy and capital cost are both negatively impacted thereby. Furthermore the evaporation plant and the stripper are completely integrated, whereby these two parts can not be independently operated.

The dimensions of the stripper also will become large, which means significant costs for the equipment.

In a conventional steam stripper also other volatile components, such as methanol, are stripped off.

Air can be used to in lieu of steam to strip the condensates. A big drawback with this method is that air is being contaminated and must be cleaned in some way. The air

volumes can also be very large. Additionally the condensate is being cooled down by the air, which has a lower wet bulb temperature as compared to the temperature of the condensate. For these reasons pure air stripping is not a realistic alternative for a modern and environmentally friendly pulp mill.

The present invention provides a possibility to strip off primarily sulfides at a very high efficiency from liquor-steam condensates from a pulp manufacturing process, and simultaneously to take care of the sulphur, thus that it will not contaminate the environment. This is being done in a closed loop concept that is comprised of three process steps, where the sulfides are stripped off from the condensate, the stripped off sulfides are being oxidized to sulphur dioxide, and to absorb the sulphur dioxide formed.

The three process steps are consequently:

1. Stripping off sulphides from liquor-steam condensate
2. Oxidation of combustible components such as sulphides and hydro carbons.
3. Absorption of sulphur dioxide.

By integrating these three process steps (1, 2, and 3) in a closed loop cycle, the cleaning of condensates can be done with a high efficiency, good heat economy, and minimal impact on the environment

The invention will in the following text be exemplified with reference to a scheme shown in the attached drawing, which schematically shows the various process steps in accordance with the invention.

In the present invention a gas is used as a medium for stripping off the sulphides from the condensate. This gas is substantially and preferably composed of air. This process step is normally designed as a scrubber column 1, where the gas 4 is introduced in the lower section and the condensate 5 in the upper section, thus that the gas and the condensate meet in counterflow contact. The contact means in the scrubber can be trays or packing material. The gas 6 leaving the scrubber will contain sulphides in form i.a. of hydrogen

sulphide and methyl mercaptan, but also organic compounds such as methanol and terpenes. This contaminated gas 6 is led to an oxidization process 2, where the gas is treated counterflow in a regenerative heat exchanger. The gas 7 from the oxidization step contains partly sulphur dioxide. These gases are then fed to a contact device, in form of a $SO_2$ scrubber 3, where the sulphur dioxide is absorbed in a preferably alkaline solution 8. The gas is then returned to the condensate scrubber to be used again as a stripping medium. In this manner is formed a closed the loop. Since oxidation in the closed loop consumes oxygen is necessary to add fresh oxygen. Additional oxygen can be added by supply 9 preferably of air or some other oxygen containing gas. The system does not allow for gas accumulation in the loop and therefore a minor portion of the gas 10 must be bled off. The gas circulation through the three process steps is accomplished by the use preferably of a fan.

Since the gas in the closed loop is primarily being circulated, an elevated level of various gas components can accumulate to rather high levels. However, since only a minor portion of the gas is bled off, the discharge of components harmful to the environment, will be limited, in spite of high concentrations in the system.

A method of improving the cleaning of the condensate in the stripper is to increase the level of $SO_2$ after the $SO_2$ scrubber (3). Such a method will result in that the condensate in the stripper (1) will get a lower pH value. A lower pH value in turn gives a better stripping of sulphides and makes possible an almost complete stripping of sulphides. This would otherwise be difficult to achieve since the condensate contains a smaller amount of alkali components, i.e. ammonia, which would increase the pH value of the condensate when the acidic sulfides are stripped off. An alkali component such as ammonia will remain in the condensate at a lowered pH. Thereby is avoided discharge of ammonia, which should otherwise be transformed to Nox, after the oxidation process.

An increase of the $SO_2$ concentration after the $SO_2$ scrubber (3) can be obtained by adjusting the supply of alkali to this stage thus that the te absorption medium will get a

comparatively lower pH. The lower the pH the higher the $SO_2$ concentration in the gas leaving the scrubber (3). The higher the $SO_2$–level in the gas, which constitutes the stripper media, the better the efficiency of stripping off sulfides from the condensate. In turn this effect can be utilized in such a way that the ratio between the condensate flow and stripper gas flow can be increased with continuos good sulphide stripping. This in turn implies an elevated level of sulphides in the stripper off gases, which in turn means an increased $SO_2$ level after the oxidization step. In this way the $SO_2$ level in the entire system can be significantly increased. This gives the following benefits the $SO_2$ concentration after the $SO_2$ scrubber can be:

1. Production of a sodiumbisulfite solution with a relative low pH is made possible.
2. The size of the plant can be reduced
3. $NO_x$ emission is reduced (see above)

The first benefit is accomplished since an increased $SO_2$ level in a gas, from an equilibrium point of view, gives a lower pH in the absorption medium. Since the addition of alkali is reduced a bisulfite solution is formed. This acid can be utilized as acidification in e.g. the bleach plant or the tall oil plant. An increased $SO_2$ –level in the recirculated gas results however in an increased $SO_2$ discharge from the system via the bleed off to the atmosphere (10). Connecting a scrubber in this point, to absorb SO2 can cure this. A scrubber in this position is preferably designed with multiple absorption steps, e.g. of the same design as the stripper. It could be so that only $SO_2$ is permitted to be absorbed in this position. In that way the $SO_2$ scrubber (3) can be eliminated from the system.

The second benefit follows the fact that the circulating gas volume substantially determines the size of the equipment. Since an increased $SO_2$ content facilitates a higher ratio of condensate/stripper gas flow, the gas flow in the system can be reduced.

The cleaned condensate will contain very low levels of sulphides and also any terpenes will be stripped off. This will give a condensate which is rather free from nasty-smelling contaminants. Methanol is another significant contaminant in black liquor condensate.

Some of the methanol will be stripped off in the stripper and some will stay in the condensate. The amount stripped off methanol is dependent on the ratio of supplied condensate to gas and the volume of the circulated gas.

The heat economy in the system is excellent since no external heat energy must be added. In the oxidation stage, heat is furthermore generated. This energy can compensate for various energy losses in the system, and any surplus can be absorbed as heat in the outgoing condensate. In other systems, where for example air is used as stripper gas, a significant amount of heat is absorbed in the air since the warm condensate transfers water vapor in contact with air. This cools down the condensate, which is avoided in the present invention, where any possible evaporated water vapor is returned to the system. It might also be possible to recover heat from the system by implementing a heat exchanger in the system. With such a heat exchanger, which cools the system, the temperature can be controlled.

There might also be a need to supply heat to the system. One reason could be to avoid oversaturated gas in certain parts of the system. As the recirculated gas, for instance after the stripper, is saturated with water vapor there is a risk that water droplets will fall out as moisture in the gas. By heating the gas, it would be possible to eliminate that moisture.

The investment costs and the size of equipment is mainly directly proportional to the amount of recirculated gas. For that reason it is important to minimize the gas recirculation. This will consequently have an impact on the methanol removal. It is therefore reasonable to count with a certain amount of methanol still remaining in the condensate. Methanol, as a pollutant in the condensate can be a drawback if the condensate is discharged to the recipient. If the condensate is being recirculated back into the process, e.g. as process water in the bleach plant, brown stock washing or limewashing, then the condensate is excellent in spite of the methanol content.

Methanol has a positive impact on bleaching, it acts as a radical scavenger and it also increases the solubility of lignin. Furthermore, this condensate is metal free. Normal process water prepared from nearby water streams always contains a certain amount of

metals, such as i.a. transition metals. These transition metals can be very harmful for the bleaching process since they decompose the bleaching agents such as hydrogen peroxide. Since the methanol act as a radical scavenger, the degradation of cellulose molecules will decrease. A metal free condensate used in the bleach plant therefore has significant benefits in spite of a certain methanol content. By recirculating the condensate to the process a discharge of oxygen consuming matters is avoided. The methanol enrichment in the process is very marginal, since the discharge of methanol from the process is relatively large for each process cycle.

The stripping of condensate can be performed in several different ways. The type of equipment chosen shall be an equipment having a very high stripper efficiency. Such type of equipment ought to have several equilibrium steps, where the condensate meets a counterflow of gas. Examples on such equipment are columns with trays or packing material. This is well defined in the technical literature, such as i.e. "Perry's Chemical Engineers' Handbook", MacGraw-Hill Book Company, 1984.

The oxidization process can be done in different ways, but the relatively low concentrations of combustible components require certain prerequisites for this type of process. A relatively high temperature is needed in order to oxidize the combustible components. A regenerative thermal oxidization process (RTO) is preferred, where the gas is treated in a heat exchanger under such temperature conditions that almost a complete oxidization takes place. Example on such a process is described in the patent application PCT/SE85/00257.

Scrubbing of the $SO_2$ gas can be done with an alkaline solution. At a pulp mill there is a surplus of alkaline process fluids. One such fluid is oxidized white liquor. In the oxidized white liquor the sulfides have been removed by oxidization. White liquor is such a strong alkali that $SO_2$ easily can be absorbed. One equilibrium stage is sufficient. A venturi scrubber is a piece of equipment wherein one equilibrium stage is almost achieved. A relatively high gas velocity can be maintained in a venturi scrubber, which makes it compact. The scrubber medium is circulated through the venturi.

The pH of the scrubber medium shall be controlled in order to control the $SO_2$ level in the gases leaving the scrubber. The venturi scrubber has also a significant benefit in that the circulating liquid can have a relatively short residence time. This implies a fast control of the pH in the scrubber. As the scrubber has only almost one equilibrium stage instead of several, a rapid response time is also achieved.

# CLAIMS

1. A method of removing sulphides and other volatile contaminants from liquor vapor condensate from a pulp manufacturing process,
characterized there in,
that the said liquor vapor condensate is fed into a stripper (1), which is part of a closed loop comprising said stripper (1) and a regenerative thermal oxidization process (RTO)(2), in which loop a gas (4), primarily consisting of air and such components formed or stripped off in the loop, is circulated, and where the circulating gas is stripping off sulphides and other volatile components from the liquor vapor condensate (5), whereafter the gas stream (6) is fed into a RTO-process (2), where the stripped off components are combusted under formation of $SO_2$, and thereafter is the $SO_2$ enriched gas (7) fed either to a $SO_2$ scrubber (3), where preferably alkali is used as absorption medium (8), whereafter the circulating gas is returned to the stripper (1).

2. A method as claimed in claim 1,
characterized in,
that the $SO_2$ scrubber (3) is part of the closed loop.

3. A method as claimed in claims 1 or 2,
characterized in,
that a minor portion of the gas (10) is bled off from the loop, at the same time air or some other oxygen containing gas (9) is supplied, to ensure that sufficient oxygen is present to safeguard that the oxidization in the RTO-process (2) takes place.

4. A method as claimed in anyone of the preceding claims,
characterized in,
that the alkali (8) used as absorption medium is oxidized white liquor.

5. A method as claimed in anyone of the preceding claims,
characterized in,
that the degree of acidification in the $SO_2$ scrubber (3) is controlled to ensure sufficient amount of $SO_2$ remaining in the gas (4) when it is returned to the stripper (1§), where $SO_2$ acidifies the condensate (5) and thereby contributes to enhance the stripping off of sulphides from the condensate.

6. A method as claimed in claim 1,
characterized in,
that a heat exchanger is installed at a suitable place in the closed loop, to recover or supply energy and thereby to control the temperature in the system.

7. A method as claimed in claim 1,
characterized in,
that the amount of recirculated gas versus the amount of condensate is controlled for the purpose of optimizing the methanol content in the condensate.

8. A method as claimed in claim 7,
characterized in,
that such condensate is used as process water in the bleach plant to reduce the bleaching chemical cost.

9. A method as claimed in claim 1,
characterized in,
that the gas (10) being bled off from the system is minimized by using pure oxygen or an oxygen enriched air mixture, necessary as make up gas (9) for the oxidization.

10. A method as claimed in claims 1 or 2,
characterized in,
that the bled off gas (10) from the system is scrubbed with regard to $SO_2$ in a separate scrubber, which preferably is made up of several absorption steps.

11. A method as claimed in anyone of the preceding claims,

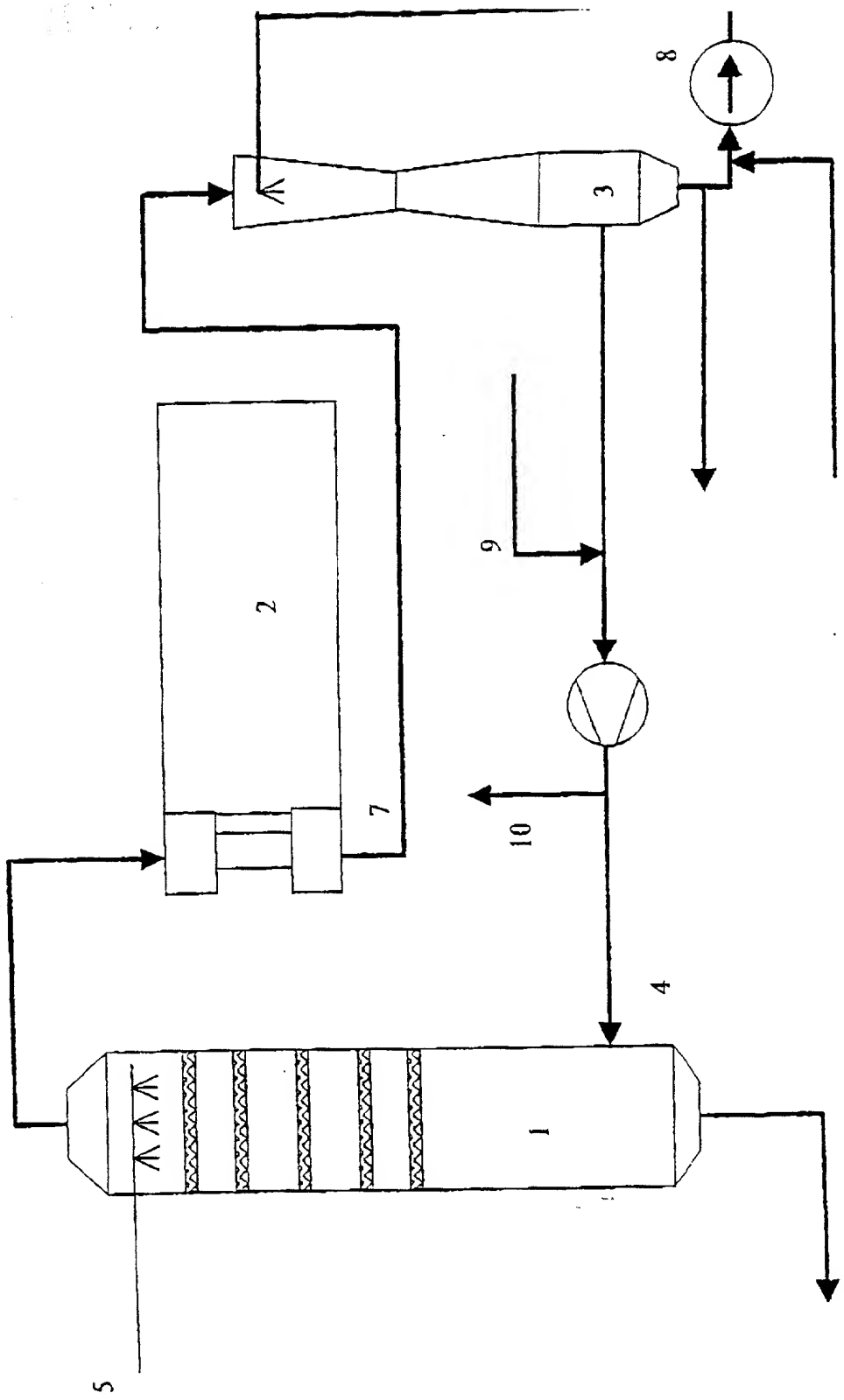
characterized in,

that the $SO_2$ level is raised to such a level in the system that the absorption medium in the $SO_2$ scrubber gets sufficient acidic, so that this fluid can be utilized as acidification agent in other areas of the pulp mill, e.g. the bleach plant or the tall oil plant.

# SUMMARY

A method to remove sulphides and other volatile contaminants from liquor vapor condensate in a pulp manufacturing process, where the mentioned liquor vapor condensate is fed into a stripper, which is part of a closed loop system comprising said stripper, a regenerative thermal oxidization process (RTO) and a $SO_2$ scrubber, in which loop a gas is circulated, preferably air and such components formed or stripped off, in this loop whereafter the circulating gas is stripping off sulphides and other volatile components from the liquor vapor condensate, whereafter the gas stream is fed into a RTO process, where the stripped off contaminants are combusted are under formation of $SO_2$ and thereafter the $SO_2$ enriched gas is led to a $SO_2$ scrubber, where preferably alkali is used as absorption medium, and thereafter the circulating gas is returned back into the stripper.
(Fig.1)

FIG 1



| **COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Includes Reference to Provisional and PCT International Applications)** | Attorney's Docket No. 010315-152 |
|---|---|

As a below named inventor, I hereby declare that:
My residence, post office address and citizenship are as stated below next to my name;
I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

A METHOD OF CLEANING SULFIDE CONTAMINATED CONDENSATES

the specification of which (check only one item below):

☐ is attached hereto.

☒ was filed as United States application
Number 09/856,961
on
and was amended
on May 29, 2001 (if applicable).

☒ was filed as PCT international application
Number PCT/SE99/02170
on November 24, 1999
and was amended
on (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 (a)-(e) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §119:

| COUNTRY (if PCT, indicate "PCT") | APPLICATION NUMBER | DATE OF FILING (day, month, year) | PRIORITY CLAIMED UNDER 35 U.S.C. §119 |
|---|---|---|---|
| Sweden | 9804061-1 | 11-26-98 | X Yes _ No |
| | | | _ Yes _ No |
| | | | _ Yes _ No |
| | | | _ Yes _ No |
| | | | _ Yes _ No |

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below.

(Application Number) (Filing Date)

(Application Number) (Filing Date)

| COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONT'D)<br>(Includes Reference to Provisional and PCT International Applications) | Attorney's Docket No.<br>010315-152 |
|---|---|

I hereby claim the benefit under Title 35, United States Code, §120 of any United States applications(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose to the Office all information known to me to be material to the patentability as defined in Title 37, Code of Federal Regulations §1.56, which became available between the filing date of the prior application(s) and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. §120:

| U.S. APPLICATIONS | | STATUS *(check one)* | | |
|---|---|---|---|---|
| U.S. APPLICATION NUMBER | U.S. FILING DATE | PATENTED | PENDING | ABANDONED |
| | | | | |
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| PCT APPLICATIONS DESIGNATING THE U.S. | | | | | |
|---|---|---|---|---|---|
| PCT APPLICATION NO. | PCT FILING DATE | U.S. APPLICATION NUMBERS ASSIGNED (if any) | | | |
| PCT/SE99/02170 | 11-24-99 | | | | |
| | | | | | |
| | | | | | |

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

| COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONT'D)<br>(Includes Reference to Provisional and PCT International Applications) | Attorney's Docket No.<br>010315-152 |
|---|---|

| FULL NAME OF SOLE OR FIRST INVENTOR<br>Kent K. SANDQUIST | SIGNATURE<br>Kent K Sandquist | DATE<br>29/8-01 |
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| POST OFFICE ADDRESS | | |
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| POST OFFICE ADDRESS | | |
| FULL NAME OF SEVENTH JOINT INVENTOR, IF ANY | SIGNATURE | DATE |
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| POST OFFICE ADDRESS | | |
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| RESIDENCE | | CITIZENSHIP |
| POST OFFICE ADDRESS | | |